

ORCHID CHEMICALS & PHARMACEUTICALS LIMITED

Factory: D-147, SIDCO Industrial Estate, Andheri - 400 119, INDIA
BATCH/FORMULA SHEET -- FURACA PREPARATION

Batch No. : ACF/201/99
 Batch Size : 91.0 Kg of 7-ACA
 Actual Output : 213.44 Kg

Time taken for
 Time: 17h 51m
 Batch Cycle Time : 27h 51m

TRIAL-1

S.No.	Raw Materials	Unit	Standard Quantity	Actual Quantity	QC Ref. No.	Lots	Checked by	Remarks
1.	Purified water for TFA	Lt	955 ± 10	945.0	2/99/147	W-1	W-1	
2.	Sodium Sulphide	Kg	54.6	515.00	QRM/1402/98	W-1	W-1	Fresh: ✓ Recovered: ✓
3.	Ethyl Acetate for TFA	Lt	635 ± 10	637.0	QMX/1013/98	W-1	W-1	
4.	Sodium Bi-carbonate	Kg	34 ± 4	34.0	QRM/1016/98	W-1	W-1	
5.	Hcl (1:1)	Lt	160 ± 15	141.0	IASU 76067	W-1	W-1	
6.	7ACA	Kg	91.0	91.0	QRM/5052/98	W-1	W-1	
7.	BF3 gas	Kg	124 ± 2	124.0		W-1	W-1	
8.	Furoyl Chloride	Kg	50.0	49.6	QRM/14233/97	W-1	W-1	
9.	Vitex - C	Kg	1.800	1.8	QRM/14983/98	W-1	W-1	
10.	Vitex - H	Kg	0.600	0.60	QRM/14983/98	W-1	W-1	
11.	Purine	Kg	1.800	1.80	QRM/5052/98	W-1	W-1	
12.	NI ₄ OH (12-15%)	Lt	220 ± 25	216.00	IASU 96064	W-1	W-1	
13.	Purified water	Lt	745 ± 10	745	2/99/147	W-1	W-1	Fresh: ✓ Recovered: ✓
14.	Ethyl Acetate	Lt	830 ± 25	820	QMX/1013/98	W-1	W-1	

Actual
 Plant in-charge

**ORCHID
 PROCESS SHEET
 ISSUED ON**

REVIEWED

PREPARED BY : PRODUCTION INCHARGE

FORMAT APPROVED BY

IN-AD PRODUCTION

HEAD PD LAB

HEAD Q.A.

ORCHID CHEMICALS & PHARMACEUTICALS LIMITED

Factory: 138 - 147, SIDCO Industrial Estate, Adampur - 601 110, INDIA

BATCH PROCESSING RECORD - RUXACA PREPARATION

Date

Batch No. : ACF 1001137
 Stage : TFA PREPARATION
 Equipment No. : GLK - 705

TRIAL - I

S.No.	Operations	Standard Quantity	Actual Quantity	Time (minutes)			Temperature °C		Remarks	Sign of Shift-Chemist / Operator
				Std	From	To	Total	Std	Actual	
1.	Check the cleanliness status of the reactor as per SOP No QAD 05 01				15:45	15:55	10			22X
2.	Charge Purified water	635 ± 5 L.	630.0		15:55	15:55	10			22X
3.	Cooling			10-20	15:45	16:00	15	22±2	20.3	22X
4.	Charge Sodium sulphide	54.6 Kg.	53.60	02-05	16:00	16:05	05	22±2	20.2	22X
5.	Stir and Give sample to Q.C. to check the sodium hydrosulphide content.			10-20	16:05	16:18	13	22±2	19.9	22X
6.	Charge Furoyl chloride slowly through GLR-705	50.0 Kg.	53.44	45-60	16:18	16:40	22	22±2	19.9	22X
7.	Stirring			05-10	16:40	17:00	20	22±2	24.3	22X
8.	Charge Ethyl Acetate	455 ± 5 L.	441.69	10-15	17:00	17:11	11	22±2	24.5	22X
9.	Stirring			02-05	17:11	17:16	05	22±2	24.8	22X
10.	Add HCl. (1:1) slowly	70 ± 5 L.	69.01	10-15	17:16	17:22	06	22±2	22.7	22X
11.	Stirring			10-15	17:22	17:35	13	22±2	22.1	22X
12.	Settling			10-20	17:35	18:15	20	22±2	21.2	22X

Plant In-charge

PREPARED BY : PRODUCTION INCHARGE : 22X

FORMAT APPROVED BY : HEAD PRODUCTION : 22X

HEAD Q.A. : 22X

HATCH PROCESSING RECORD - FURACA PREPARATION

Batch No. : ACF / 001 / 199
 Stage : TFA PREPARATION
 Equipment No. : GLR - 709

Date :

S.No.	Operations	Standard Quantity	Actual Quantity	Time (minutes)			Temperature °C		Remarks	Sign of Shift Chemist / Operator
				Std	From	To	Std	Actual		
13.	Separate the Aq. layer in MLI-702	-	-	20-30	18:55	19:35	22±2	20.5		
14.	Charge Purified water to Ethyl acetate layer.	320 ± 5 L.	320.01	10-15	18:35	18:45	22±2	20.8		
15.	Add sodium Bicarbonate to adjust the pH.	34 ± 4 Kg	34.04	30-45	18:45	19:40	22±2	20.8	pH should be 7.0 - 7.2 Observed pH: 7.05	
16.	Stirring	-	-	20-30	19:45	20:15	22±2	20.8		
17.	Settling	-	-	10-15	20:15	20:30	22±2	20.8		
18.	Separate lower aqueous layer in carboys	-	-	10-15	20:30	20:40	22±2	20.9	Collected Aq. Layer in clean carboys Volume of aq. Layer: 276 L.	
19.	Send Ethyl Acetate layer for recovery	-	-	10-15	20:40	20:45	22±2	20.9	Volume of Ethyl acetate: 391 L.	
20.	Clean the reactor as per SOP No. PRD.07.0311	-	-	10-20	20:45	20:55	22±2	21.7		
21.	Check the cleanliness status of the reactor as per SOP No. QAD.05.01	-	-	05-10	20:55	21:05	22±2	22.0		
22.	Charge aqueous layer of step No. 18	-	-	15-20	21:05	21:20	22±2	22.7		
23.	Charge Ethyl Acetate	180 ± 5 L.	180.0	10-15	21:20	21:30	22±2	23.4	pH should be 0.9 - 1.0 Actual pH: 0.85	
24.	Add HCl (1:1) slowly	90 ± 5 L.	11.01	10-20	21:30	21:45	22±2	23.7		
25.	Stirring	-	-	10-15	21:45	21:55	22±2	23.7		
26.	Settling	-	-	10-15	21:55	22:10	22±2	25.3		
27.	Separate lower aqueous layer	-	-	20-30	22:10	22:50	22±2	25.3	Vol. of aqueous layer: 116.5 L.	
28.	Cool the Ethyl acetate Layer	-	-	10-20	22:50	23:10	12±2	-		
29.	Collected Ethyl Acetate layer in clean carboys and check the M/C	-	-	15-25	23:10	23:20	12±2	25.3	Vol. of Ethyl Acetate: 270.0 L. M/C: %	
30.	Clean the reactor as per SOP No. PRD.07.0311	-	-	10-20	23:20	23:40	-	-		

20. The layer separation must be done very carefully. There should not be any free water in EA layer.

Plant In-charge

Shift In-charge

BATCH PROCESSING RECORD - FURACA PREPARATION

Batch No. : ACF 1601199
Stage : CONDENSATION
Equipment No. : GLR - 701

Sl. No.	Operations	Standard Quantity	Actual Quantity	Time (minutes)			Temperature °C		Remarks	Sign of Shift Chemist / Operator
				Std	From	To	Std	Actual		
31.	Check the cleanliness status of reactor as per SOP No. : PRD 0501	-	-	20-40	14.30	14.40	RT	-	-	MA
32.	Charge Ethyl Acetate and check the M/C.	365 ± 5 Lt.	365 Lt.	45-60	14.40	15.00	52	29	M/C should be Not more than 0.5% Observed: 0.053	MA
33.	Cool under nitrogen	-	-	180-300	15.00	15.50	3-12	-	Make sure the BF3 scrubber is on. The ending temp. must be 11 ± 1°C	MA
34.	Purge NF3 gas	124 ± 2 Kg	124.0	02-05	15.50	20.00	-	10.2	-	MA
35.	Charge Vilex C	1.8 Kg	1.8	-	20.00	20.05	11 ± 1	09.7	-	MA
36.	Increase the temperature. (if required)	-	-	05-10	23.10	23.15	11 ± 1	10.2	-	MA
37.	Charge T-ACA	91.0 Kg	91.0	05-10	23.15	23.25	11 ± 1	10.2	The reaction is exothermic	MA
38.	Charge TFA (Ethyl acetate Layer from step No.29)	240 ± 20 Lt.	240.0	-	23.25	23.25	30 ± 1	-	-	MA
39.	Warm up using hot water	-	-	130-240	23.25	03.05	30 ± 1	30.4	TACA content should not be more than 1%	MA
40.	Slit and Give sample for HPLC in every 60 minutes	-	-	10-25	03.05	03.15	30 ± 1	29.4	-	MA
41.	Transfer for hydrolysis	-	-	-	03.15	03.20	-	-	-	MA
42.	Flushing of reactor with Ethyl acetate and transfer to Hydrolysis reactor.	5-10 Lt.	10.0	10-20	03.20	03.40	-	-	-	MA
43.	Clean the reactor as per SOP No: PRD-07-0311	-	-	-	03.40	20	-	-	-	MA

REACTION MONITORING DATA

Sl. No.	After Hour	HPLC Time	TACA NMT 1.0%	ACF NLT 80.0%	TPA NMT 10.0%	OI NMT 2.0%
1	60	60 min	5.91%	84.92	8.04%	1.20
2	120	120 min	2.92%	90.06	4.84	2.16
3	180	150 min	1.69	91.07	2.84	4.40
4	240	180 min	0.88	94.54	2.67	1.90

MA
Plant In-charge

Shift In-charge

BATCH PROCESSING RECORD - FURACA PREPARATION

Batch No. : ACF 1001/45
 Stage : HYDROLYSIS
 Equipment No. : GLR - 709

Date :

S.No.	Operations	Standard Quantity	Actual Quantity	Time (minutes)			Temperature °C		Remarks	Sign of Shift Chemist / Operator
				Std	From	To	Std	Actual		
44.	Check the cleanliness status of the reactor as per SOP No. QAD05.01	-	-	-	02.00	02.15	-	-		<i>Chibhan</i>
45.	Charge Purified Water	275 ± 10 L.	275.0	10-15	02.15	02.30	-	-		<i>Chibhan</i>
46.	Cool	-	-	20-30	02.30	03.00	3±1	3.9		<i>Chibhan</i>
47.	Add Vitex - H	0.600 Kg.	0.60	01-02	03.00	03.02	3±1	3.9		<i>Chibhan</i>
48.	Receive the condensation mass and add purine	1.800 Kg.	1.80	-	03.02	03.25	05-20	13.7	Final temperature will go to 15-20°C	<i>Chibhan</i>
49.	Add NH ₄ OH solution (15-20%)	195 ± 245 L.	216	30-120	03.25	5.10	20±1	19.7	pH should be 3.45-3.55 Actual pH: 3.61	<i>Chibhan</i>
50.	Stir	-	-	30-35	5.10	05.40	20±1	20.0		<i>Chibhan</i>
51.	Check the pH and readjust (if required)	-	-	-	05.35	05.40	-	20.0	pH should be 3.45-3.55 Actual pH: 3.60	<i>Chibhan</i>

Sh
 Shift In-charge

Prkash
 Plant In-charge

BATCH PROCESSING RECORD - FURACA PREPARATION

Batch No. : ACF1001/99.
 Stage : CENTRIFUGING & MILLING
 Equipment No. : ADM-CF-20 / GNR-701

Date :

S.No.	Operations	Standard Quantity	Actual Quantity	Time (minutes)			Temperature °C		Remarks	Sign of Shift Chemist / Operator
				Std	From	To	Std	Actual		
52.	Check the cleanliness status of the centrifuge. QAD05.02	-	-	-	05:25	05:40	-	-		<i>[Signature]</i>
53.	Feed slurry to centrifuge	-	-	60-120	05:40	06:10	2011	20:5	Operate the centrifuge as per SOP No: PRD.070101	<i>[Signature]</i>
54.	Spin under nitrogen	-	-	30-60	06:10	06:40	-	-		<i>[Signature]</i>
55.	Clean the reactor as per SOP No: PRD.07.311	-	-	-	06:40	06:45	-	-		<i>[Signature]</i>
56.	Spray washing with Ethyl acetate through reactor.	90 ± 5 Lt	200.0	10-20	06:45	06:55	-	-		<i>[Signature]</i>
57.	Spin under nitrogen	-	-	60-90	06:55	07:30	-	-	Quantity of M/L: 9.60 Lt	<i>[Signature]</i>
58.	Unload the material in double polybags.	-	-	-	07:30	09:15	-	-		<i>[Signature]</i>
59.	Clean the centrifuge as per SOP No: PRD.07.0301	-	-	10-20	09:15	09:35	-	-		<i>[Signature]</i>
60.	Check the cleanliness status of the multi mill as per SOP: No.QAD.05.03.	-	-	10-20	09:35	09:45	-	-		<i>[Signature]</i>
61.	Mill the wet cake using 5 mm mesh	-	-	60-90	09:45	09:45	-	-	Operate the multi mill as per SOP No: PRD.07.01.04	<i>[Signature]</i>
62.	Clean the multi mill/ Granulator as per SOP No: PRD.07.0304	-	-	20-30	09:45	10:05	-	-		<i>[Signature]</i>

Wet Weight : 338.05 Kgs

[Signature]
 Plant In-charge

[Signature]
 Shift In-charge

BATCH PROCESSING RECORD - FURACA PREPARATION

Page 6 of 6

Batch No. : ACF/061199
 Stage : SLURRY WASHING OF CAKE
 Equipment No. : GLR-209/CF-701/ MM-1/ GNR-

Date

S.No.	Operations	Standard Quantity	Actual Quantity	Time (minutes)			Temperature °C		Remarks	Sign of Shift Chemist / Operator
				Std	From	To	Std	Actual		
1)	Check the cleanliness status of the reactor as per SOP No. QAD05.01	-	-	-	08.35	09.40	-	-	-	1006
2)	Charge Ethyl acetate	270 ± 5	275	10-15	09.40	09.55	RT	25	-	1006
3)	Charge Purified Water and cool	270 ± 5	275	10-15	09.55	10.00	RT	25	-	1006
4)	Charge wet material	-	338.05 Kg	05-10	10.00	10.35	22±2	20.2	-	1006
5)	Stirring	-	-	15-20	10.35	11.00	22±2	21.7	-	1006
6)	Check the cleanliness status of the centrifuge. QAD05.02	-	-	-	11.00	11.30	-	-	-	1006
7)	Feed slurry to centrifuge	-	-	30-60	11.30	12.00	22±2	21.8	Operate the centrifuge as per SOP No: PRD.070101	1006
8)	Spin under nitrogen atmosphere	-	-	30-45	12.00	12.30	-	-	-	1006
9)	Spray washing with Ethyl acetate through reactor.	100.00 Li	100.00	10-15	12.30	12.45	-	-	-	1006
10)	Spin under nitrogen atmosphere	-	-	20-30	12.45	13.05	RT	-	Quantity of Mfr.: 90 Li	1006
11)	Spray wash with purified water through reactor	100.00 Li	100.00	10-15	13.05	13.10	RT	-	-	1006
12)	Spinning	-	-	30-40	13.10	13.30	RT	-	-	1006
13)	Spray wash with purified water through reactor.	100.00 Li	100.00	10-15	13.30	13.55	RT	-	-	1006
14)	Spinning	-	-	90-120	13.55	15.05	RT	-	-	1006
15)	Clean the reactor as per SOP No: PRD.07.311	-	-	-	15.05	15.25	-	-	-	1006
16)	Unload the material in double polythene bags.	-	-	30-45	15.25	15.35	-	-	-	1006
17)	Clean the centrifuge as per SOP No: PRD.07.0301	-	-	10-20	15.35	15.45	-	-	Multimill the material if required	1006
18)	Check the cleanliness status of the multi mill as per SOP: No. QAD.05.03.	-	-	10-20	15.45	15.55	-	-	-	1006
19)	Multimill the wet cake using 5 mm mesh	-	-	60-90	15.55	16.45	-	-	Operate the multi mill as per SOP No: PRD.07.01.04	1006
20)	Clean the multi mill as per SOP No: PRD.07.0804	-	-	20-30	16.45	17.25	-	-	-	1006

20: The material should be packed under nitrogen and stored below 10°C

Wet Weight : 868.49 Kgs

Shift In - charge

Plant In - charge

ORCHID CHEMICALS & PHARMACEUTICALS LIMITED

Factory: 100-142, SIDCO Industrial Estate, Andheri - 400 110, INDIA

BATCH FORMULA SHEET - EXTRACTION OF FLUORIDES FROM ML

Time: 18:00 Hr.

Time: 0935 Hr.

TRIAL-1

Batch No. : ACF/ 001/99
 Batch Size : 91.0 Kg of 7-ACA
 Actual Output : 1185 L.

Starting Date
 Completion Date
 Batch Cycle Time : 15 hrs 25 min

S.No.	Raw Materials	Unit	Standard Quantity	Actual Quantity	QC Ref. No.	Lots Checked by	Remarks
1.	Volume of ACF ML and washing	LL	2300±100	1680	-	APM	
2.	Purified water	LL	55±10	5450	C/09154	APM	
3.	Caustic Lye	LL	20±5	180	QRM/1119/98	APM	

P. Bahadur
 Shift In-charge

APM
 Plant In-charge

PREPARED BY : PRODUCTION INCHARGE : APM

HEAD TO LAB:

HEAD Q.A.: 08/12

FORMAT APPROVED BY

HEAD PRODUCTION:

[illegible]

Batch No. _____
 Stage _____
 Equipment No. _____

: ACF / 001 / 99
 : Excretion of Fluorides from batch ML
 : SSR - 706

BATCH PRO

AGE 190/30V

ACF / 001 / 94)

TRIAL 1

S.No.	Operations	Standard Quantity	Actual Quantity	Time (minutes)				Temperature-°C		pH		Remarks	Sign of Shift Chemist / Operator
				Std	From	To	Total	Std	Actual	Std	Actual		
1.	Check the cleanliness status of the reactor as per SOP No QAD-05.01.					17:40	18:40	30					R
2.	Charge mixture of Mf. and all EA and water washing	1580.1				18:00	18:30	30					R
3.	Stirring			10-15		18:30	18:45	15	22±2	23.5			R
4.	Settling			10-15		18:45	19:00	15	22±2	23.5		Check fluoride in aqueous layer Observed: 570.00ppm	R
5.	Separate the layers			10-15		19:00	19:10	10	22±2	23.5			R
6.	Transfer aqueous layer to SSR-713			10-15		19:10	19:25	15	22±2	23.5			R
7.	Charge water to organic layer	185 Lt.	185	02-05		19:25	19:30	5	22±2	23.8	7.0-7.5		R
8.	Adjust pH by adding caustic lye solution	15-20 Lt.	17.2	10-15		19:30	19:45	15	22±2	24.00	7.5		R
9.	Stirring			20-25		19:45	20:10	25	22±2	24.0			R
10.	Settling			10-15		20:10	20:40	30	22±2	24.0		Check fluoride in aqueous layer Observed: 171.00ppm	R
11.	Separate the layer			10-15		20:40	20:50	10	22±2	24.2			R

Prakash

Ques.
Plant In - charge

PRODUCTION IN CHARGE: Akman.

FORMAT APPROVED BY

HEAD PRODUCTION:

HEAD PD LAB:

HEAD O.A.:

BATCH PROCESSING RECORD - EXTRACTION OF FLUORIDES FROM ML

Page 1 of 3

Date :

Batch No. : ACT / 00 1 / 99
 Stage : Extraction of Fluoride from Batch ML
 Equipment No. : SSR-706

S.No.	Operators	Standard Quantity	Actual Quantity	Time (minutes)			Temperature °C		pH		Remarks	Sign of Shift Chemist / Operator
				Std	From	To	Total	Std	Actual	Std		
12.	Transfer aqueous layer to SSR-713	-	-	10-15	20.50	21.05	15	22±2	21.5			R
13.	Charge water to organic layer	185.0 Lt.	185 Lt.	02-05	21.05	21.10	5	22±2	22.0			R
14.	Adjust pH by adding caustic lye solution	05-10 Lt.	0 Lt.	10-15	21.10	21.25	15	22±2	22.0	7.0-7.5	7.5	R
15.	Stirring	-	-	20-25	21.25	21.50	25	22±2	22.0			R
16.	Settling	-	-	10-15	21.50	22.10	20	22±2	22.5		Check fluoride in aqueous layer	W
17.	Separate the layer	-	-	10-15	22.10	22.20	10	22±2	22.5		Observed: 12.8 ppm	W
18.	Transfer aqueous layer to SSR-713	-	-	10-15	22.20	22.30	10	22±2	22.5			W
19.	Charge water to Organic layer	185 Lt.	185 Lt.	02-05	22.30	22.35	05	22±2	22.6	7.0-7.5	7.41	W
20.	Adjust pH by adding caustic lye solution (if required)	-	1.0 Lt.	10-15	22.35	22.40	05	22±2	22.7		Observed: 24.642	W
21.	Stirring	-	-	20-25	22.40	22.50	20	22±2	22.7			W
22.	Settling	-	-	10-15	22.50	23.15	15	22±2	22.9			W
23.	Separate the layer	-	-	10-15	23.15	23.30	15	22±2	22.9		Check fluoride in aqueous layer	W
24.	Transfer aqueous layer to SSR-713	-	-	10-15	23.30	23.40	10	22±2	22.9		Observed: 21.5 ppm	W
25.	Ethyl acetate layer in drums and sent to recovery of ethyl acetate	-	-		23.40	23.51	15	22±2	22.9		Volume of Ethyl Acetate Layer:	W

Operator :
 Plant In-charge

Shift In-charge

BATCH PROCESSING RECORD - EXTRACTION OF FLUORIDES FROM ML

Batch No. : ACF / 001 / 87
Stage : Recovery of Fluoride from Aqueous Layer
Equipment No. : SSR - 713

S.No.	Operations	Standard Quantity	Actual Quantity	Time (minutes)			Temperature °C		pH		Remarks	Sign of Shift Chemist / Operator
				Std	From	To	Std	Actual	Std	Actual		
26.	Check the cleanliness status of reactor as per SOP No. : PRD 0501				16:50	17:20	20±2	22.0				
27.	Check the receiving of aqueous solution from step 6, 12, 18 & 24				18:00 20:00 22:00 23:15	19:05 21:05 23:30 24:00	20±2	22.1				
28.	Adjust the pH by caustic lye (If required)	60-70 Li.	52.08	30-40	3:35	4:05	20±2	22.4	7.0-7.5	7.11	Ag. lye = 60 lbs	
29.	Stirring (Check pH)			15-20	4:05	4:05			7.0-7.5	7.10		
30.	Heat to distill out ethyl acetate upto 85°C vapour temperature			180-300	4:25	09:15	Upto 95°C	95			Ethyl acetate recovered: 95.68 Out : A85 MC-522	
31.	Pump to PVR -714 for fluoride recovery				09:25	09:35					Residual volume: 1200	

Note : The residual solution after Ethyl acetate recovery "SHOULD NOT" be pumped to MPDIJ area tanks. It has to be recovered in Cefazolin plant only.

P. Kalu: *[Signature]*

Shift In-charge

[Signature]
Plant In-charge

ORCHID CHEMICALS & PHARMACEUTICALS LIMITED

Factory: 138 - 147, SIDCO Industrial Estate, Alibour, INDIA

BATCH FORMULA SHEET - CEFOTIOXIM HYDROCHLORIDE

Batch No. : CFCL/001/99
 Batch Size : 91.0 Kg of 7-ACA
 Standard Output : 150.0 - 160.0 Kg.
 Actual Output : 154.40 Kg

TRIAL - 1

Time: 15.45 Hr.
 Time: 16.55 Hr.
 Standard Yield : 1.643 - 1.758 Kg/Kg of 7-ACA
 Actual Yield :
 Batch Cycle Time : 1.696 hr.
 37.45 hr.

S.No.	Raw Materials	Unit	Standard Quantity	Actual Quantity	QC Ref. No.	Lots Checked by	Remarks
1.	Furac wal	Kg.	-	262.49	QCFP/1002/99	BSR	
2.	MAEM	Kg.	127.4	127.4	QMA/1012/1013/99	BSR	
3.	Aminothane	Kg.	53.20	53.2	QRM/5492/98	BSR	
4.	Purine	Kg.	4.500	4.5	QRM/5032/98	BSR	
5.	Vivex	Kg.	4.500	4.5	QRM/4983/98	BSR	
6.	Ethyl acetate	Li.	1050.00	1050.0	QRM/1283/98	K. Ran.	
7.	THF	Li.	2180.00	2180	QRM/3140/98	K. Ran.	
8.	Sodium Chloride	Kg.	211.00	211	QRM/5962/98	K. Ashok	
9.	HCl - 35 - 37 % (LR grade)	Li.	100.00	41 + 57	QRM/1111/98	K. Ran.	
10.	Eno carbon	Kg.	18.00	18.0	QRM/1075/98	BSR	
11.	Compound PCA	Kg.	0.100	6.120	QRM/3353/98	BSR	
12.	Iso propyl Ether	Li.	515.00	455 + 120	QMS/1010/02	BSR	
13.	Acetone	Li.	300.00	300	QMS/1010/02	BSR	
14.	Purified Water	Li.	1000 - 1050	936	C/99153	K. Ran.	

ORCHID
 PROCESS SHEET
 ISSUED ON

Plant In-charge

PREPARED BY: PRODUCTION INCHARGE: Q. Ran.

FORMAT APPROVED BY:

HEAD PRODUCTION: V. Ran.

HEAD Q.A.: P. Ran.

REVIEWED

ORCHID CHEMICALS & PHARMACEUTICALS LIMITED
 Factory: 138 - 147, SIDCO Industrial Estate, Abhinav, INDIA
BATCH PROCESSING RECORD - CEFTIOFUR HYDROCHLORIDE

Batch No. : CFCL/001/99
 Stage : CONDENSATION
 Equipment No. : GLR - 304

TRIAL - 1

Date

S.No.	Operations	Standard Quantity	Actual Quantity	Time (minutes)			Temperature °C		Remarks	Sign of Shift Chemist / Operator
				Std	From	To	Std	Actual		
1.	Check the cleanliness status of the reactor as per SOP No. QAD05.01	-	-	-	15.45	16.00	-	-	-	BE-R
2.	Charge THF	700.00 Lt.	700.85	10-20	16.40	16.20	-	-	-	BE-R
3.	Charge FURACA Wet	-	262.49 Kg	10-15	19.30	19.45	-	9.0	From 91 Kg. FURACA ⇒ 16°C. Invert 22 by meter • (455 - (Net weight of Furaca - 109)) { 495 - (662 - 49 - 109) }	BE-R
4.	Charge purified Water (* see Remarks)	-	301.54 Lt.	10-15	19.00	19.15	-	-	-	BE-R
5.	Cooling by using -25°C brine	-	-	120-150	17.00	19.00	4±1	5.0	Already Jacketed Reactor 25°C brine	BE-R
6.	Charge MAEM	127.400 Kg	127.400	10-15	19.50	20.00	4±1	4.8	-	BE-R
7.	Flush with THF	30.0 Lt.	30.84	10-15	20.00	20.15	4±1	4.5	-	BE-R
8.	Add Anisothane	53.2 Kg	53.84	45-60	20.15	21.25	4±1	4.0	pH should be between 9.0-9.2 Observed pH: 9.18	BE-R
9.	Stir and Give sample for reaction monitoring	-	-	240-300	21.25	01.20	4±1	4.2	-	K-Ran

Indicates critical process / operation parameter

REACTION MONITORING

S.No.	Actual Time	Reaction Monitoring Minutes	% of Furaca	% of CFCL	% of MAEM	% of NMT	% of OI
1.	23.25	120	1.35	76.25	7.54	12.74	0.78
2.	00.25	180	0.91	77.92	6.00	12.16	1.18
3.	01.10	240	0.89	77.17	6.62	12.41	0.56
4.		300	NMT				
		Standard limit	1.0%				

Shift In-charge

Plant In-charge

PREPARED BY: PRODUCTION INCHARGE: QKwal

FORMAT APPROVED BY: QKwal

HEAD PRODUCTION: QKwal

HEAD Q.A.: QKwal

BATCH PROCESSING RECORD - CEFTIOXIME HYDROCHLORIDE

Date :

Batch No. : CPCL/001/99
 Stage : EXTRACTION
 Equipment No. : GLR - 303,304,307

S.No.	Operations	Standard Quantity	Actual Quantity	Time (minutes)			Temperature °C		Remarks	Sign of Shift Operator
				Std	From	To	Std	Actual		
10.	Charge Ethyl Acetate and simultaneously raise the temperature using CI water	685.0 Lt.	685	15-20	01-20	01-35	16	15.0		K. Ra.
11.	Add Purine	4.500 Kg.	4.5	02-05	01-35	01-38	16	15.1		K. Ra.
12.	Add Vitex	4.500 Kg.	4.5	02-05	01-38	01-40	16	15.2		K. Ra.
13.	Stirring	--	--	15-20	01-40	02-00	16	15.2		K. Ra.
14.	Settling	--	--	20-30	02-00	02-20	16	15.8		K. Ra.
15.	Check the cleanliness status of the reactor GLR-307 as per SOP No. QAD05.01	--	--	--	02-15	02-20	--	--		K. Ra.
16.	Separate the layer and collect the aqueous layer in GLR-307	--	--	10-15	02-20	02-35	16	15.8	Volume: 710 Lt.	K. Ra.
17.	To the aqueous layer, charge ethyl Acetate in GLR-307	360.00 Lt.	360	10-15	02-35	02-50	16	15.8		K. Ra.
18.	Stirring	--	--	15-20	02-50	03-05	19	16.4		K. Ra.
19.	Settling	--	--	15-20	03-05	03-25	16	16.4		K. Ra.
20.	Check the cleanliness status of the reactor GLR-303 as per SOP No. QAD05.01	--	--	05-10	03-20	03-35	--	--		K. Ra.
21.	Separate the layer from GLR-307. Collect the aqueous layer to GLR-303	--	--	45-60	03-35	04-05	10	16.7	Volume: 650	K. Ra.
22.	Check the cleanliness status of the reactor GLR-304 as per SOP No. QAD.05.01	--	--	--	--	--	--	--	Not Required	K. Ra.
23.	Transfer the Ethyl acetate layer to GLR-304	--	--	10-20	04-05	04-15	10	16.8		K. Ra.

Shift in-charge

Plant in-charge

BATCH PROCESSING RECORD - CEFTIOXUR HYDROCHLORIDE


Page 3 of 7

Batch No. : CFCL/001199
 Stage : 1st EXTRACTION
 Equipment No. : GLR - 304

Date :

S.No.	Operations	Standard Quantity	Actual Quantity	Time (minutes)				Temperature °C		Remarks	Sign of Shift Chemist / Operator
				Std	From	To	Total	Std	Actual		
24.	Charge purified Water in Ethyl Acetate layer in GLR - 304	180.0 L.	180	15-20	04-05	04-20	15	16±1	16.4		K. Rao
25. 20	Stirring	--	--	15-20	04-20	04-35	15	16±1	16.6		K. Rao
26.	Settling	--	--	15-20	04-35	04-50	15				K. Rao
27.	Separate the aqueous layer and transfer to GLR-303	--	--	30-40	04-50	05-10	20				K. Rao
28.	Transfer the Ethyl acetate layer to PRC - 322	--	--	30-45	05-30	11-00	30			Volume: 1620 L of FA+THF Layer.	K. Rao


 Shift In-charge


 Plant In-charge

BATCH PROCESSING RECORD - CEFTIOFUR INDIROCHLORIDE

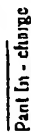
Page 4 of 7

Batch No. : CFCL/001199
 Stage : III Extraction and Charcolisation
 Equipment No. : GLR - 303, 307

Date :

S.No.	Operations	Standard Quantity	Actual Quantity	Time (minutes)			Temperature °C		Remarks	Sign of Shift Chemist / Operator
				Std	From	To	Std	Actual		
29.	Charge purified water in the combined aqueous layer in GLR-303	455.00 L.	455	10-20	4.15	4.25	2022	18.2		K. Ran.
30.	Stirring	--	--	05-10	04.25	4.30	2022	18.0		K. Ran.
31.	Charge THF	1250.00 L.	1250	15-20	04.30	4.50	2022	18.2	Control the temperature - 25°C time slowly	K. Ran.
32.	Add Sodium Chloride	211.00 Kg	211.0	15-20	04.50	05.05	2022	18.3		K. Ran.
33.	Stirring	--	--	15-20	05.05	05.25	2022	18.3		K. Ran.
34.	Add conc. HCl to adjust the pH	45 L.	41	30-45	05.25	05.50	2022	18.4	pH should be 2.9-3.0 observed pH: 2.9	K. Ran.
35.	Stirring	--	--	10-15	05.50	06.05	2022	18.6	pH collection to 30 with pH probe	K. Ran.
36.	Settling	--	--	30-40	06.05	07.00	2022	18.6		K. Ran.
37.	Separate the aqueous layer and collect in PRC-323	--	--	30-45	07.00	07.45	2022	18.4	Volume of aqueous layer: 1668.10 L.	K. Ran.
38.	Charge Endo carbon in THF layer	18.0 Kg	18.0	02-45	07.45	07.50	2022	18.6		K. Ran.
39.	Stirring	--	--	30-45	07.50	08.30	2022	19.0		K. Ran.
40.	Check the cleanliness status of the sparkler filter as per SOP No. QAD.05.05	--	--	05-10	08.30	08.30	2022	--		K. Ran.
41.	Filter the solution through SF - 307/302 and collect the filtrate in GLR-307	--	--		08.30	09.30	2022	19.5	Operate the sparkler filter as per SOP No. PRD.03.0103 Volume = 1550 L.	K. Ran.
42.	Flush the reactor with THF at the end of filtration over and collect the filtrate through SF-307/302 in GLR - 307	200.00 L.	200.0		09.30	09.45	2022	19.8	Clean the sparkler filter as per SOP No. PRD.03.0204 Volume = 1750 L.	K. Ran.
43.	Clean the reactor as per SOP. No. PRD.03.0208 GLR-303.	--	--		09.50	10.00	--	--		K. Ran.


 Shift In-charge


 Plant In-charge

BATCH PROCESSING RECORD - CEFTHIOXIME HYDROCHLORIDE

Date :

Batch No. : CFCI.1 001/99
 Stage : CRYSTALLIZATION
 Equipment No. : GLR - 307

S.No.	Operations	Standard Quantity	Actual Quantity	Time (minutes)			Temperature °C		Remarks	Sign of Shift Chemist / Operator
				Std	From	To	Total	Std	Actual	
44.	Check the cleanliness status of the reactor-GLR-307 as per SOP No. QAD05.01	-	-	-	08.15	08.20	05	-	-	<i>[Signature]</i>
45.	Collect the filtrate in GLR-307	-	-	-	08.30	09.45	75	19±1	19.8	<i>[Signature]</i>
46.	Add 35 % Conc. LR Grade to bring down the pH	54.0 L.	54	30.40	09.45	10.28	43	19±1	20.0	<i>[Signature]</i>
47.	Add compound PCA	100.0 gm.	100	01.02	10.28	10.30	02	19±1	18.2	<i>[Signature]</i>
48.	Stirring	-	-	45.60	10.30	11.30	60	19±1	18.0	<i>[Signature]</i>
49.	Add IPTG slowly	455.00 L.	455	45.60	11.30	12.30	60	19±1	19.0	<i>[Signature]</i>
50.	Stirring	-	-	15.20	12.30	12.45	15	19±1	19.1	<i>[Signature]</i>
51.	Cool using -25°C brine	-	-	-	12.45	13.30	45	2±2	04	<i>[Signature]</i>
52.	Stirring	-	-	45.60	-	-	-	2±2	-	<i>[Signature]</i>
53.	Check the % of CFCI. Content in AGL.	-	-	-	13.30	16.20	170	2±2	1.1	<i>[Signature]</i>
CFCI should be NMT : 0.2 % Observed : 0.22 %										

[Signature]
 Shift In-charge

[Signature]
 Plant In-charge

BATCH PROCESSING RECORD - CENTRIFUGAL HYDROCHLORIDE

Batch No. : CFCL/ 001/99
 Stage : CENTRIFUGING & MILLING
 Equipment No. : CF-301& MM- 301, 303

Date :

S.No.	Operations	Standard Quantity	Actual Quantity	Time (minutes)			Temperature °C		Remarks	Sign of Shift Chemist / Operator
				Std	From	To	Std	Actual		
54.	Check the cleanliness status of the Centrifuge SOP NO. QAD05.02	--	--	--	16.00	16.15	--	--	--	K.R.
55.	Feed the slurry to the centrifuge	--	--	75-90	16.20	18.20	212	1.0	Operate the Centrifuge as per SOP No. PRD.03.0101	K.R.
56.	Spinning	--	--	45-60	18.00	19.20	204.5	--	--	K.R.
57.	Charge IPE in the reactor GLR-307 and cool it.	130.0 L.	130.0 L.	--	18.25	19.20	191.1	19.5	--	K.R.
58.	Spray wash with IPE	130.0 L.	130.0 L.	10-15	19.20	19.40	191.1	19.5	Volume of IPE + THF: 2250 L.	K.R.
59.	Spinning	--	--	45-60	19.40	20.40	--	--	--	K.R.
60.	Spray wash with acetone with reduced RPM of centrifuge	150.00 L.	150.00 L.	15-20	21.00	21.30	--	--	--	K.R.
61.	Spinning at mode 2 RPM	--	--	15-20	21.30	22.15	--	--	--	K.R.
62.	Spray wash with acetone with mode 1 reduced RPM	150.00 L.	150.00 L.	15-20	22.15	23.00	--	--	--	K.R.
63.	Spinning at mode 2 RPM	--	--	100-120	23.00	02.00	--	--	--	K.R.
64.	Unload the material from the centrifuge	--	--	45-60	03.00	04.00	--	--	MC Checked after this run MC: 14.50% 19.52%	K.R.
65.	Clean the centrifuge as per SOP No. PRD 03.0201	--	--	--	04.00	04.15	--	--	--	K.R.
66.	Check the cleanliness status of the Multimill as per SOP No. QAD05.03	--	--	--	04.15	04.20	--	--	milling stopped, as material not in use	K.R.
67.	Mill the wet cake	--	--	60-70	04.20	05.20	--	--	Operate the Multimill as per SOP No. PRD03.0102	K.R.
68.	Clean the Multimill as per SOP No. PRD03.0203	--	--	--	05.20	05.30	--	--	--	K.R.

WET WEIGHT BEFORE DRYING: 225.6 Kg 99.6 +126.0

Shift In-Charge

Plant In - charge

(Signature)

BATCH PROCESSING RECORD - CENTRIFUGAL HYDROCHLORIDE

Batch No. : CFCL/001/99
 Stage : CENTRIFUGING & MILLING
 Equipment No. : CF - & MM - 303

Date :

S.No.	Operations	Standard Quantity	Actual Quantity	Time (minutes)			Temperature °C		Remarks	Sign of Shift Chemist / Operator
				Std	From	To	Std	Actual		
54.	Check the cleanliness status of the Centrifuge SOP NO. QAD05.02	--	--	--	--	--	--	--		
55.	Feed the slurry to the centrifuge	--	--	75-90	--	--	242	--	Operate the Centrifuge as per SOP No. PRD.03.0101	
56.	Spinning	--	--	45-60	--	--	2045	--		
57.	Charge IPE in the reactor GLR-3U7 and cool it.	130.0 Lt.	--	--	--	--	1911	--		
58.	Spray wash with JPE	130.0 Lt.	--	10-15	--	--	1911	--	Volume of IPE + JPE :	
59.	Spinning	--	--	45-60	--	--	--	--		
60.	Spray wash with acetone with reduced RPM of centrifuge	150.00 Lt.	--	15-20	--	--	--	--		
61.	Spinning at mode 2 RPM	--	--	15-20	--	--	--	--		
62.	Spray wash with acetone with mode-1 reduced RPM	150.00 Lt.	--	15-20	--	--	--	--		
63.	Spinning at mode 2 RPM	--	--	100-120	--	--	--	--		
64.	Unload the material from the centrifuge	--	--	45-60	--	--	--	--		
65.	Clean the centrifuge as per SOP No. PRD.03.0201	--	--	--	--	--	--	--		
66.	Check the cleanliness status of the Multimill as per SOP No. QAD05.03	--	--	--	--	--	--	--		
67.	Mill the wet cake	--	--	60-70	4-20	5-20	--	--	Operate the Multimill as per SOP No. PRD03.0102	
68.	Clean the Multimill as per SOP No. PRD03.0203	--	--	--	--	--	--	--		

WET WEIGHT BEFORE DRYING : _____ Kg.

Shift In-charge

Plant In-charge

BATCH PROCESSING RECORD - CEFTIONUR HYDROCHLORIDE

Date :

Batch No. : CFCL/001/99

Stage : DRYING

Equipment No. : VTD - 302

Wet Weight : 99.6 Kg.

PART - 1

Specified hot water temperature in the dryer : 40 - 45°C

S.No.	Operations	Time (minutes)			Vacuum mm/Hg	Temp.	Remarks	Sign of Shift Chemist / Operator
		Slid	From	To	Total			
1.	Check for cleanliness status of the dryer as per SOP No. QAD05.04	--	05.20	05.25	05			K. Ra.
2.	Charge the material in the dryer	45 - 60	05.25	06.00	35			K. Ra.
3.	Apply vacuum	65 - 70	06.00	09.15	95		Operate the Dryer as per SOP No. PRD 03.0104	K. Ra.
4.	Apply hot water	--	07.15	07.45	1530		Make sure the specified temperature.	for
5.	Draw sample after 240 min. of drying for checking MC (See Remarks)	340 - 600	04.00	08.45	1605		MC should not be more than 5.0 %	for
	Note down vacuum at regular interval of 60 min.	0	06.00	06.00	0		Observed MC : 3.7	K. Ra.
		60	06.00	06.00	60	7.00		K. Ra.
		120	06.00	06.00	120	7.00	41.0	K. Ra.
		180	06.00	06.00	180	7.00	41.0	K. Ra.
		240	06.00	06.00	240	7.00	41.0	K. Ra.
		300	06.00	06.00	300	7.00	41.0	K. Ra.
		360	06.00	06.00	360	7.00	42.0	K. Ra.
		420	06.00	06.00	420	7.00	42.0	K. Ra.
		480	06.00	06.00	480	7.00	43.0	K. Ra.
		540	06.00	06.00	540	7.00	43.3	K. Ra.
		600	06.00	06.00	600	7.00	44.0	K. Ra.
		45 - 60	08.45	09.45	60		67.34 kg (9.45 - 25.15)	for
6.	Unload the material							for
7.	Clean the dryer as per SOP No: PRD.03.0205	05 - 10	09.45	09.55	10			for

Break the vacuum by nitrogen slowly.

Total Dry Weight : 67.34 Kg.

Shift In-charge

Plant In-charge

BATCH PROCESSING RECORD - CERTIOFULHYDROCHLORIDE

Date :

Batch No. : CECL / 001 / 99
 Stage : DRYING
 Equipment No. : VTD - 803
 Wet Weight : 126.0 Kg

Specified hot water temperature in the dryer : 40 - 45°C

S.No.	Operations	Time (minutes)			Vacuum mm/Hg	Temp.	Remarks	Sign of Shift Chemist / Operator
		Std	From	To				
1.	Check for cleanliness status of the dryer as per SOP No. QAD05.04	-	5.25	5.30	05			K. Rao
2.	Charge the material in the dryer	45.60	5.30	6.00	80			K. Rao
3.	Apply vacuum	65.70	6.00	7.15	75		Operate the Dryer as per SOP No. PRD 03.0104	K. Rao
4.	Apply hot water	-	7.15	16.00	515	-	Make sure the specified temperature.	As per specification
5.	Draw sample after 240 min. of drying for checking MC (Sec Remarks)	540 - 600	6.00	16.00	600	-	MC should not be more than 5.0 %	As per specification
	Note down vacuum at regular interval of 60 min.	0	6.00	6.00	0	-	Observed MC : 3.9%	As per specification
		60	6.00	7.00	60	700		As per specification
		120	6.00	8.00	120	700	41.0	As per specification
		180	6.00	9.00	180	700	41.0	As per specification
		240	6.00	10.00	240	700	41.0	As per specification
		300	6.00	11.00	300	700	42.0	As per specification
		360	6.00	12.00	360	700	42.0	As per specification
		420	6.00	13.00	420	700	42.0	As per specification
		480	6.00	14.00	480	700	43.0	As per specification
		540	6.00	15.00	540	700	43.1	As per specification
		600	6.00	16.00	600	700	44.0	As per specification
6.	Unload the material	45.60	16.00	16.45	45	-		As per specification
7.	Clean the dryer as per SOP No; PRD.03.0205	05.10	16.45	16.55	10	-		As per specification

205 Break the vacuum by nitrogen slowly.

Dry wt. 87.06

Total Dry Weight : 154.4 Kg.

Shift In-charge

Plant In-charge

DEVIATION REPORT - FORM A

Date:

Name of the product: CEFTIOFUR HYDROCHLORIDE Batch No.: CFU/001/99

Deviated Operation & parameter : Addn. of Aminothane & Time

BPR page No.: 1 of 7 Serial No.:

Standard Value : 45-60 mts Actual Value: 70 mts

Reason for Deviation : From Receiver PRC 312 bottom valve and G9
30A Receiving valve kept open fully but it took that much
time

Chemist / Operator : [Signature]

Shift In charge : [Signature]

ANALYSIS

Occurrence : ☐ First time ☐ Rarely ☐ Frequently

Corrective Action taken / planned These were three batches with new process
where input may take some deviate time. Based on
findings from #1 to 7 we will fix the specification.
(New process furnace was used). The addition line
system will be checked again.

[Signature]
Head-Production

Comments (Criticality on the Yield/ Quality of Output)

Not critical

[Signature]

Head / In charge PD Lab

Material Disposition : ☒ Accepted as is ☐ Reprocessed ☐ Rejected

Approved by :

[Signature]
Quality Assurance

Date:

Effective Date :